



Experimental investigation on shrinkage and surface replication of injection moulded ceramic parts

Islam, Aminul; Giannakas, Nikolaos; Marhöfer, David Maximilian; Tosello, Guido; Hansen, Hans Nørgaard

Published in:
Proceedings of the 4th International Conference on Nanomanufacturing (nanoMan2014)

Publication date:
2014

[Link back to DTU Orbit](#)

Citation (APA):
Islam, A., Giannakas, N., Marhöfer, D. M., Tosello, G., & Hansen, H. N. (2014). Experimental investigation on shrinkage and surface replication of injection moulded ceramic parts. In *Proceedings of the 4th International Conference on Nanomanufacturing (nanoMan2014)*

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Experimental investigation on shrinkage and surface replication of injection moulded ceramic parts

A. Islam*, N. Giannakas, D. M. Marhöfer, G. Tosello, H. N. Hansen

Department of Mechanical Engineering, Technical University of Denmark, Produktionstorvet, Building 427A,
DK-2800 Kgs. Lyngby, Denmark

**corresponding author (email: mais@mek.dtu.dk)*

ABSTRACT:

Ceramic moulded parts are increasingly being used in advanced components and devices due to their unprecedented material and performance attributes. The surface finish, replication quality and material shrinkage are of immense importance for moulded ceramic parts intended for precision applications. The current paper presents a thorough investigation on the process of ceramic moulding where it systematically characterizes the surface replication and shrinkage behaviours of precision moulded ceramic components. The test parts are moulded from Catamold TZP-A which is Y_2O_3 -stabilised ZrO_2 having widespread uses in the advanced industrial applications. The influence of the sintering process on the achievable roughness and replication differences between the mould surface and the final parts are discussed based on the experimental findings. Moreover, an investigation on the morphology and porosity distribution for the moulded ceramic parts is presented.

Keywords: Ceramic Moulding, Sintering, Surface analysis, Shrinkage.

1 Introduction

Ceramic Injection Moulding (CIM) is a net-shape manufacturing process which combines plastic injection moulding and performance attributes of ceramic materials. Ceramic has been a material of choice for demanding parts that require high resistance to heat, corrosion, wear and calls for bio-compatibility. Shape complexity, dimensional accuracy, replication fidelity, material variety etc. combined with high-volume capabilities are some of the key advantages of CIM process. The market for ceramic moulding is growing at a speed of 15 to 20 % per year [1] and has already found many applications in the micro manufacturing especially in the area of medical technology, micro-mechanics, biosensors, microsystem and in microfluidics [2]. Further research and thorough understanding about the surface replication, shrinkage behaviours and effects of the process conditions on the final part quality will open up exciting application areas for CIM. The aim of the current paper is to experimentally characterize the shrinkage behaviours, surface replication and achievable tolerance of the injection moulded ceramic parts. The effects of physical location of the moulding gate on shrinkage and surface quality are also studied.

2 Materials and methods

The test geometry used for the experiment was a simple flat disc with diameter of 8 mm and thickness of 1 mm. The material chosen for this ceramic injection moulding experiments was Catamold TZP-A, a commercially available material from BASF, Germany. Fig 1 shows the test geometry and Catamold material used in the experiment. Catamold is ready-to-mould granules for the production of sintered ceramic components in polycrystalline yttria-stabilised tetragonal zirconia [3]. Current investigation is done to test the feasibility of producing precision components for high end engineering application- for example the component production for hearing aid applications. The selected materials for hearing aids should generate excellent surface finish, should be capable of producing the finest details and should combine wear resistance with high strength. The chosen Catamold material has already been used successfully in the area of biomedical, micro engineering, fibre optics and in nozzles applications [3]. This suggests that Catamold TZP-A has the potential to be a successful material for hearing aid application and this was the motivation to choose the material.



Disc part with the sprue, runner and gate Standard granulates of Catamold TZP-A
Fig. 1: Test geometry and Catamold TZP-A material used in the experiment.

The machine used for moulding the test samples was an Engel ES 80/25 HL-Victory type machine with a screw diameter of 18 mm and clamping force of 25 tons. Moulding of the parts was done with the standard process condition recommended by the material supplier. During the preliminary screening study based on the design of experiment technique, it was found that the Catamold material was very sensitive to the process parameter variations. Successful moulding and demoulding of the ceramic parts could be achieved in extremely narrow process window. In particular, moulding trial showed that successful parts could only be produced with the process conditions recommended by material supplier. This fact hindered the realization of the initial DOE plan and parts were moulded only with the parameters listed in the material data sheet given in reference [3].

The parts were moulded using two different mould inserts having two different surface roughnesses (average roughness S_a 2.53 and 3.38 μm respectively for insert 1 and insert 2). The roughness transfer from the mould inserts to the green parts and finally from the green parts to the sintered parts was systematically characterized. Both the green and sintered parts were analysed for roughness, shrinkage and morphological investigations. The part after moulding and before sintering is termed as green part and part after sintering is called sintered part.

The debinding and sintering processes were performed at Formatec Ceramics BV, Goirle, The Netherlands. After moulding, debinding and sintering the parts were ready for metrological investigations. The roughness measurement was done with Alicona Infinite Focus Microscope. For coordinate measurement an optical coordinate measuring machine (Optical CMM- Demeet 220) was used. For morphological and microscopic investigations a scanning electron microscope (SEM- JEOL JSM-5000) and an optical microscope (Olympus GX 41) were used. The roughness of the mould inserts, green parts and sintered part were measured in three different locations based on the distance from the moulding gate: close to gate (cg), in the middle (mg), and far

from the gate (fg). Afterwards, the results were compared to investigate the roughness transfer throughout the process chain at various part locations.

3 Results and discussion

3.1 Disc diameter and part shrinkage

To investigate the shrinkage of the CIM parts, the disc diameter was measured both on the green and sintered parts. The instrument used for this measurement was Demeet 220 optical CMM. In Fig 2, the results of average diameter measurements for green and sintered parts are presented in comparison with the measurements taken on the mould cavity. The average was taken from 8 different sample measurements. It shows excellent cavity-to-cavity reproducibility and a significant post-sintering shrinkage of the disc diameter.

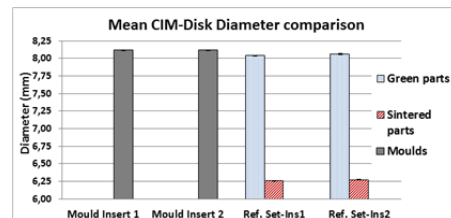


Fig. 2: Disk diameter comparison of green and sintered parts moulded with mould insert 1 and 2.

The shrinkage of the green parts after moulding was relatively low which was about 0.97-0.66 %, but for the sintered part the shrinkage was about 22 % (presented in plot-A of Fig 3). The thicknesses of the green and sintered parts were also measured using the same Optical CMM. The shrinkage found in the thickness of the sintered parts was about 21 %. The comparative shrinkage of the disc thickness and diameter are presented in the Fig 3 (plot-B). It shows the difference in shrinkage in different direction of the moulded part is not significant; meaning the shrinkage of ceramic part is almost isotropic. But this is not the case for all powder moulding situations. For example as reported in reference [4], when parts are moulded with metal powder, after sintering the final parts tend to shrink more in the thickness direction. Current experiment shows this is not the case for ceramic moulding situation. The difference in shrinkage behaviour for metal and ceramic is attributed by the weight difference of the two materials. Heavy metal particles influence the shrinkage toward the gravity direction and increase the thickness shrinkage. On the other hand ceramic particles are much lighter compared to the metal particles and this actually helps the ceramic parts to achieve relatively uniform shrinkage in all directions.

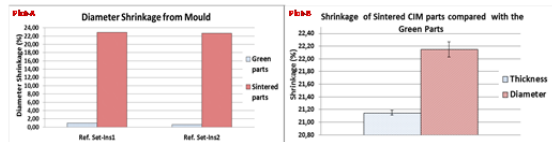


Fig. 3: Comparison of diameter shrinkage of green and sintered parts with respect to measured mould diameter (plot-A), comparison of thickness shrinkage and diameter shrinkage of the sintered ceramic parts (plot-B).

3.2 Surface replication and part roughness

Roughness measurement was performed according to ISO 4288 standard. The scan area of the roughness measurement was $1.25 \times 0.22 \text{ mm}^2$ and a 50 times magnification lens was used to scan the surface. An L_c : 0.25 mm roughness filter was used to retain the uniformity of the measurements. Roughness measurements were conducted at three positions, close to gate, in the middle of the part and far from the gate as mentioned before. The surface roughness values of S_a (arithmetic average roughness) and S_z (average distance between five highest peaks and lowest valleys) were investigated. Fig 4 shows the comparative surface topography of mould insert, green part and sintered part. The pictures were taken in the middle of the parts.

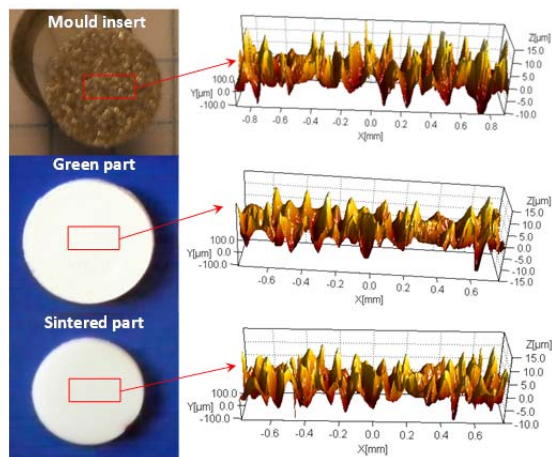


Fig. 4 Comparative surface topography of the mould (insert 2), green part and sintered part made from the same insert.

When the roughness of the green and sintered parts is compared, it is evident that sintering smoothens the surface (observed in all roughness parameters). Fig 5 shows the S_a roughness values where measurements were taken in the middle position of the mould inserts, green parts and sintered parts. It is clear from the plot that the parts produced with insert 2 have larger roughness (S_a green: 3.35 µm , sintered: 3.08 µm) simply because

the mould insert have higher roughness values. It is to be noted that parts made with insert 2 get closer to the average mould roughness compared with the parts made with insert 1. The average roughness (S_a) of the sintered parts made with insert 1 decrease considerably to 20 % whereas the decrease of S_a of the parts made with insert 2 is only 8 %.

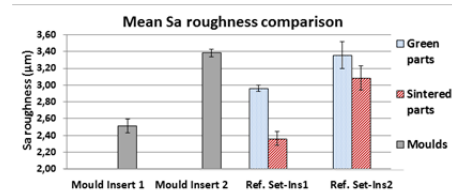


Fig. 5 Comparison of S_a roughness parameters among mould inserts, green parts and sintered parts.

The roughness deviations of green and sintered parts compared with the mould roughness were examined for both inserts. The measurements reveal an interesting relation between the mould topography and the surface replication of the parts. The green parts made with insert 2 replicated better compared to the parts produced with mould insert 1. The surface of insert 2 was better replicated due to a uniform distribution of valleys and peaks and due to their larger size, allowing better filling and compaction of the powder material. This is confirmed by lower surface shrinkage of the roughness parameters in parts produced with mould insert 2 compared with the roughness parameters measured on the parts made with insert 1 (the comparison is presented in Fig 6-plot A).

When the reduction in roughness parameters with respect to mould values is taken into consideration, the sintered parts of insert 1 exhibit a slightly smaller reduction in S_a (6 %) value, in comparison with sintered parts from insert 2 where the reduction for S_a is 8.7 % (presented in Fig 6-plot B). When the reduction of S_z values are considered, a different behaviour is visible; the value of sintered parts in comparison to mould values exhibit a significant reduction- 36 % for the part produced with insert 1 and 12 % for the part produced with insert 2. The larger reduction of S_z values indicates worse replication of roughness parameters in case of insert 1 as the peaks and valleys of insert 1 are not replicated correctly. Conclusion from this observation is, the final parts from mould insert 2 are better replicated as the sintered parts in comparison with green parts and mould inserts, exhibit similar roughness trend and small levels of roughness reduction.



Fig. 6 Roughness reduction of the sintered parts compared with the green parts (plot-A), roughness reduction of the sintered parts compared with the mould surface (plot-B).

Surface roughness at different sections of the parts

The comparison of surface roughness at different sections of the part was done. Different sections were chosen based on the proximity from the moulding gate. The mould inserts had slightly higher surface roughness in the surface laying far from the gate. This trend of surface roughness has been replicated quite well by the ceramic moulding except from the middle section of the parts made with insert 1. Fig 7 shows the surface replication of the CIM part at various locations. This indicates that a customize surface within the same ceramic part is possible to generate.

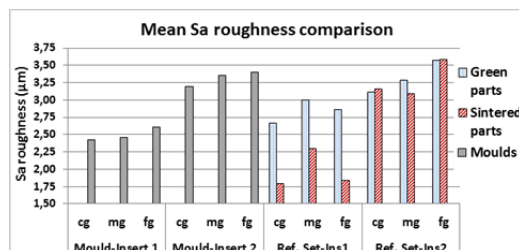


Fig. 7: Sa roughness comparison of green and sintered parts moulded with mould insert 1 and 2, at different locations: close to gate (cg), middle of the part (mg) and far from gate (fg).

3.3 Morphological investigation

For morphological investigation, the surface and the polished cross section of the green and sintered parts were investigated under SEM and optical microscope. Fig 8 shows the SEM micrographs of the surface of the parts made by mould insert 1 and 2. The comparison between the green and sintered parts clearly shows smoothening effect of the sintering process. Surface roughness of the sintered parts is decreased for both mould inserts. The surface porosities are visible in the sintered parts (black spots in the right side pictures of Fig 8).

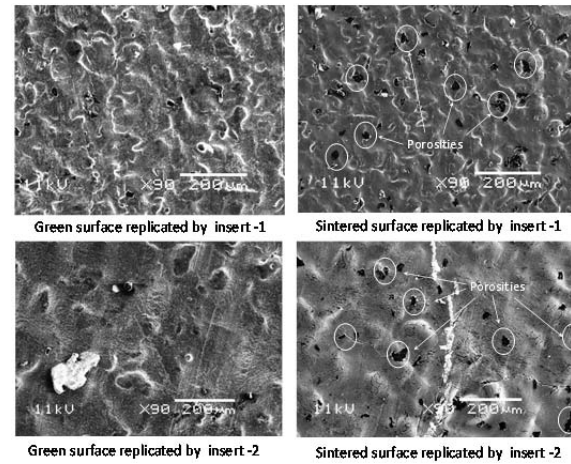


Fig. 8: Surface of green and sintered parts produced from mould insert 1 and 2- surface pores are visible on the sintered part surface (some pores are indicated with the white circles).

Microscopic investigations at different part locations (like close to the gate, in the middle and far from the gate), showed no significant difference in the porosity distribution for the final parts. This shows the uniformity of the part quality moulded by CIM process but in few cases large pores or voids were visible in the cross sections of the CIM parts. The reasons for these types of porosities are not clearly understood. It can be the impurities in the ceramic granulate or it can be the non-homogeneous mixture of ceramic particles with polymer binder.

4 Future work

Future work will focus on possible ways to run experiments for characterizing the effects of different moulding conditions on the shrinkage and surface replication of the ceramic parts. The experiments should be repeated with other grades of ceramic feedstock. A comparison of shrinkage and surface replication quality between ceramic and metal feedstock for micro moulding is very interesting and this issue will be addressed in future research.

5 Conclusion

This paper investigated the surface properties and shrinkage behaviour of injection moulded ceramic parts. The mould shrinkage of the Catamold material observed was about 1 % for the green parts. For the final sintered part the shrinkage in the radial direction was 22 % (diameter shrinkage) and in the thickness 21 %. This fact indicates that the shrinkage difference in different direction of the ceramic parts was not significant. The relatively lighter ceramic particles do not increase the thickness shrinkage as it can be the case with the metal injection moulding [4]. Interesting observations were made with the surface roughness of the parts. Experimental observations showed that more accurate reproducibility of the mould was possible with coarse surface, meaning higher roughness could be replicated well by the process chain of ceramic moulding and sintering. For the accurate reproduction of the mould surface by CIM process, the uniform distribution of surface peaks and valleys proved to be important.

In general, the sintering process had some smoothing effect on the surface. The surface roughness was decreased after sintering. But the study showed different roughness from various locations of the mould surface transferred proportionately to the final sintered parts. The morphological investigation showed quite uniform porosity distribution. No significant difference was observed in the porosity distribution of the sintered parts based on the distance from the moulding gate. This indicated that the moulded ceramic parts were quite homogeneous in terms of porosity distribution.

6 Acknowledgments

This paper reports work undertaken in the context of the project “HiMicro- High Precision Micro Production Technologies”. HiMicro is a Collaborative Research Project supported by the European Commission in the 7th Framework Programme (FP7-2012-NMP-ICT-FoF: 314055). Formatec Ceramics BV, The Netherlands, is acknowledged for their support for sintering and supply of the Catamold TZP-A raw material.

7 References

- [1] R. G. Cornwall; R. M. German, “Powder Injection Molding – World Markets and Technologies”, Center for Innovative Sintered Products, Pennsylvania State University, University, Euro PM, 2004.
- [2] M. Sutter, “Micro-injection molding puts ceramics in top form”, Mikroproduktion, Carl Hanser Verlag, München, 2009.
- [3] BASF SE Catamold ® TZP-A-Processing Instructions, BASF, VH/CA 004 e, July 2008.
- [4] A. Islam; H.N. Hansen; N.M. Esteves; T.T. Rasmussen, “Effects of holding pressure and process temperatures on the mechanical properties of moulded metallic parts”, part of: ANTEC 2013 Conference Proceedings, 2013, Presented at: ANTEC 2013, Cincinnati, OH 2013.